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(54) **DIESEL FUEL COMPOSITIONS
CONTAINING LEVULINATE ESTER**

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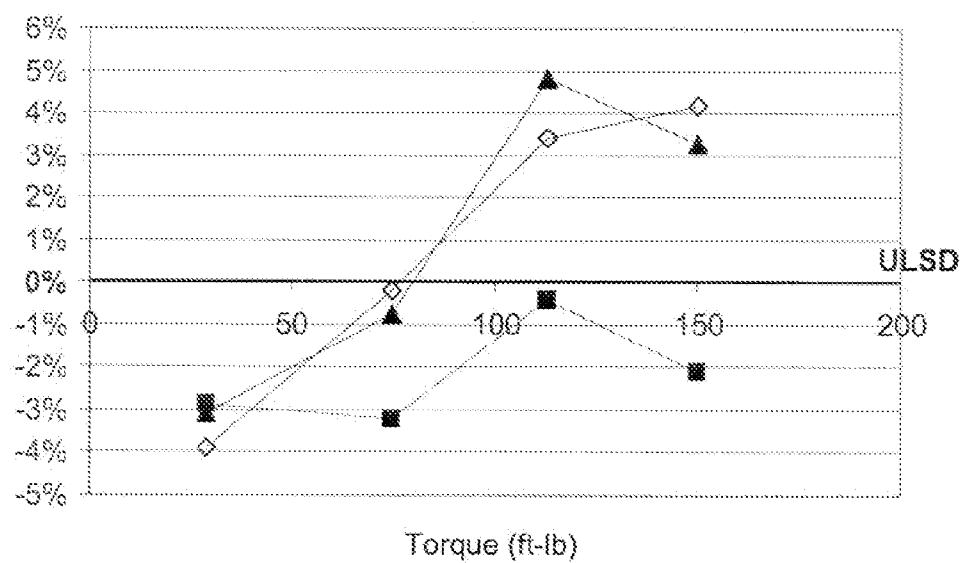
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(57) **ABSTRACT**

A diesel fuel composition is disclosed that requires a reduced amount of petroleum-based diesel and provides lower level of emissions, while maintaining the desired performances of the petroleum-based diesel. The disclosed fuel composition comprises a petroleum-based diesel and at least about 5% by volume of levulinate ester, and may be substantially free of alkanolamides, alcohols, or combinations thereof. When desired, the fuel composition may include biodiesel and other additives. The disclosed fuel composition may be formulated such that its performances are in compliance with the ASTM D975 standard.

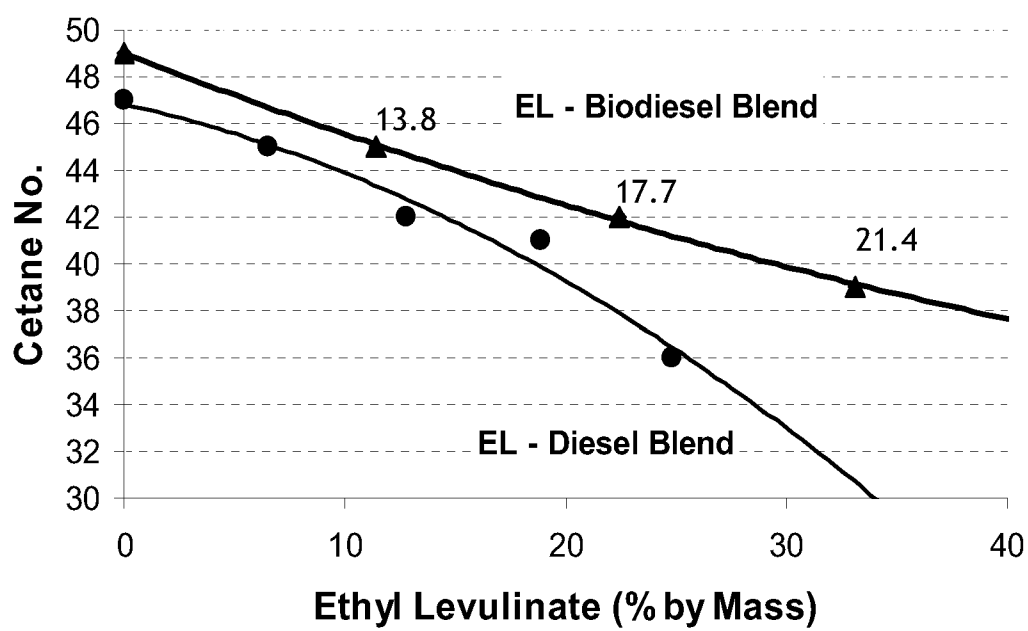
FIGURE 1

% Change in
Fuel Consumption



◇ EL5
■ B20
▲ BEL20

FIGURE 2



DIESEL FUEL COMPOSITIONS CONTAINING LEVULINATE ESTER

[0001] This non-provisional application relies on the filing date of provisional U.S. Application Ser. No. 61/187,305 filed on Jun. 16, 2009, having been filed within twelve (12) months thereof, which is incorporated herein by reference, and priority thereto is claimed under 35 USC §1.19(e).

BACKGROUND OF THE DISCLOSURE

[0002] Diesel fuel has continued to be a critical fuel for growth around the world due to its cost and availability. A continuing increase in the global demand for fuels and the limited non-renewable petroleum supply has led to higher fuel prices. The combustion of diesel fuel in engines emits pollutants such as carbon-based particulates, hydrocarbons, nitrogen oxide, and carbon monoxide. The growing environmental concerns have led to the use of ultra low sulfur diesel fuels, commonly referred to as diesel fuels having 50 ppm sulfur or less ("ULSD fuels"). As of 2007, almost every diesel fuel available in America and Europe is the ULSD type. This ULSD diesel has poor fuel lubricity, and therefore, causes excessive wear of fuel injectors and other moving parts that come in contact with the fuel. In the United States, diesel fuel must comply with the American Society for Testing and Materials Standard (ASTM) D975 protocol, which describes a limited number of properties that diesel fuels must meet. This ASTM standard specifies only the performance related requirements demanded of a fuel for a diesel engine, and it does not mandate the composition of the fuel. Some of these regulated properties are flash point, water and sediment content, kinematic viscosity, ash content, sulfur content, carbon residue, lubricity, and distillation temperature. Over the past decade, the fuel technologies have been focused on increasing the diesel fuel efficiency and reducing the emitted pollutants.

[0003] Several approaches have been used to reduce the pollutant emission. The common approach is to replace a portion of hydrocarbons in diesel fuel with other organic compounds that provide cleaner exhaust emissions and do not adversely affect engine performance. The widely used organic compounds for blending with the diesel to provide cleaner exhaust emissions are alcohols, such as methanol and ethanol. For example, a blend of 15% ethanol and 85% diesel oil provides a diesel fuel of acceptable burning capacity without the necessity of modifying existing diesel engines. However, one major drawback of this approach is that ethanol and methanol are immiscible with diesel fuel within the normal range of operating temperatures. Therefore, the fuel blend cannot be uniformly mixed or blended into one phase without rapid separation into their individual components. The separation of diesel and alcohol is further accelerated by water present in the diesel and alcohol.

[0004] To address the immiscibility problem, other ingredients must be added into the diesel/alcohol blend to solubilize and stabilize it as a homogenous fuel blend. Surfactants have been used to enhance the miscibility. U.S. Pat. No. 6,695,890 discloses the use of a surfactant additive comprising fatty acid diethoxylamide, an alcohol ethoxylate, and an ethoxylate of a fatty acid to stabilize the diesel/alcohol blend. U.S. Pat. No. 7,357,819 teaches an additive for a diesel/alcohol blend, comprising 2-ethylhexanol, fatty alkanolamides, and fatty acids. U.S. Publication No. 2008/0110081

describes a diesel fuel composition that is substantially free of alkanolamides and contains (i) at least 95% by volume of a diesel fuel; (ii) 0.1%-5% by volume of levulinic acid or a functional derivative thereof; and (iii) 0.1%-5% by volume of an additive selected from: (a) alkoxyated monoalcohols, (b) polyols, (c) alkoxyated fatty acids, and (d) ethoxyated dimeric fatty acids. Biodiesel may optionally be included in the fuel composition. U.S. Publication No. 2009/0013591 discloses a diesel fuel composition including: (i) a diesel fuel/alcohol blend containing about 15%-95% by volume of diesel fuel and about 10%-85% by volume of one or more C1-10 alcohols; and (ii) about 1-15% by volume of the glycerol ether or mixture of glycerol ethers, based on the fuel alcohol blend volume.

[0005] Extensive effort has been focused on replacing some or all petroleum-based diesel fuel with a fuel derived from renewable sources. Biodiesel refers to a variety of ester-based oxygenated fuels made from raw materials such as vegetable oils, animal fats, used cooking oil, and waste from pulp and paper industry. Biodiesel has higher cetane number, viscosity, and Cloud point compared to the conventional petroleum-based diesel fuels. U.S. Publication No. 2005/0268535A1 discloses a biodiesel fuel blend including a biodiesel fuel and an additive capable of reducing particulate emissions and improving fuel economy while improving engine performance of the biodiesel. However, due to its higher Cloud point and Pour point temperatures compared to conventional diesel fuel, biodiesel may lead to engine starting problems in cold weather. Moreover, Biodiesel may not be compatible with certain engine parts such as fuel pumps, fuel lines, and valves, and thereby causing a premature failure of the parts. To address these drawbacks, blends of diesel/biodiesel have been developed that retain the conventional diesel performances. For example, the "B20" fuel is a blend 80% petroleum-based diesel and 20% biodiesel (by % volume) that is recognized as an alternative fuel under the Energy Policy Act (EPACT) in the United States. U.S. Pat. No. 5,578,090 discloses a diesel blend of (i) about 25%-95% by wt of diesel fuel and (ii) 5%-75% by wt of the additive that comprises 10%-75% by wt of fatty acid alkyl esters, 1%-25% by wt of glyceryl ethers, and 0%-10% by weight of triglycerides, wherein the alkyl group on the ethers and the esters is selected from the group consisting of C1-10 straight, branched or cyclic alkanes. However, in general, the diesel/biodiesel blends suffer from several inferior characteristics compared to the conventional petroleum-based diesel, such as increased level of NOx emissions and higher gelling temperature. U.S. Publication No. 2005/0160663 discloses the use of fuel borne catalyst (FBC) as an additive for the blend of biodiesel and a low aromatic content ULSD having less than 10% aromatics. The FBC additive is made of a fuel-soluble metal selected from the group consisting of platinum, iron, cerium, and the combinations thereof. However, the FBC catalyst additive is non-renewable and quite costly.

[0006] Accordingly, there is still a need for diesel fuel compositions suitable for the existing diesel combustion engines and in compliance with the ASTM D975 standard that retain the desired fuel characteristics of petroleum-based diesel such as Cloud Point and Pour Point temperatures, cetane number, fuel efficiency and lubricity; while reducing the required amount of petroleum-based diesel and lowering the emissions of pollutants such as unburned hydrocarbons, particulate matters, carbon monoxide, and NO_x gases.

[0007] Further, it is desirable that the components in the fuel compositions, besides the petroleum-based diesel, are economical and derived from renewable sources.

SUMMARY OF THE DISCLOSURE

[0008] A diesel fuel composition is disclosed that requires a reduced amount of petroleum-based diesel and provides lower level of emissions, while maintaining the desired performances of the petroleum-based diesel. The disclosed fuel composition comprises a petroleum-based diesel and at least about 5% by volume of levulinate ester, and may be substantially free of alkanolamides, alcohols, or combinations thereof. When desired, the fuel composition may include biodiesel and other additives. The disclosed fuel composition may be formulated such that its performances are in compliance with the ASTM D975 standard.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009] FIG. 1 is a graph showing, when the engine was run at a speed of 2800 rpm and different torque values, percentage changes in the fuel consumption of different fuel blends: EL5 (a blend of 95% by volume of ULSD and 5% by volume of ethyl levulinate), B20 (a blend of 80% by volume of ULSD and 20% by volume of biodiesel), BEL20 (a blend of 80% by volume of ULSD, 13.3% by volume of biodiesel, and 6.7% by volume of ethyl levulinate), and ULSD (ultra low sulfur diesel) fuel; and

[0010] FIG. 2 is a graph showing an effect on the cetane numbers of the fuels upon addition of ethyl levulinate at different levels.

DETAILED DESCRIPTION OF THE DISCLOSURE

[0011] The present disclosure now will be described more fully hereinafter, but not all embodiments of the disclosure are necessarily shown. While the disclosure has been described with reference to exemplary embodiments, it will be understood by those skilled in the art that various changes may be made and equivalents may be substituted for elements thereof without departing from the scope of the disclosure. In addition, many modifications may be made to adapt a particular situation or material to the teachings of the disclosure without departing from the essential scope thereof.

[0012] The diesel fuel composition of the present disclosure comprises a petroleum-based diesel and at least about 5% by volume of levulinate ester based on total volume of the fuel composition. When desired, the disclosed fuel composition may further include biodiesel.

[0013] In one embodiment of the present disclosure, the diesel fuel composition comprises:

[0014] (a) from about 60% to about 80% by volume of the petroleum-based diesel;

[0015] (b) from about 5% to about 15% by volume of the biodiesel; and

[0016] (c) at least about 5% by volume of the levulinate ester.

[0017] In one embodiment, the disclosed fuel composition is substantially free of alkanolamides, alcohols, or combinations thereof. The term "substantially free" refers to less than 1% by volume of such component in the disclosed fuel composition.

[0018] Various petroleum-based diesel fuels may be used in the present disclosure. These include, but are not limited to,

fuel oil NO. 2 or NO. 4 petroleum distillates; syntroleum (diesel fuel range derived from Flscher-Tropsch synthesis); petroleum-based diesel fuels containing emulsified water or alcohols such as ethanol or methanol; ultra low sulfur fuels (ULSD) such as diesel fuel containing less than 0.05% sulfur, and blends thereof.

[0019] A variety of levulinate esters may be used in the present disclosure. These include, but are not limited to, methyl levulinate, ethyl levulinate, butyl levulinate, glycol ester of levulinic acid, and blends thereof. In one embodiment, the disclosed diesel composition includes ethyl levulinate. In one embodiment, the disclosed diesel composition includes glycol ester of levulinic acid.

[0020] When desired, the disclosed fuel composition may further comprise one or more additional additives to further enhance selected fuel performance. These additives may include, but are not limited to, detergents, octane boosters, cetane number improvers, metal deactivators, corrosion inhibitors, antioxidants, rust inhibitors, dispersants, biocides, and combinations thereof.

[0021] In one embodiment of the present disclosure, the diesel fuel composition comprises:

[0022] (a) from about 60% to about 80% by volume of the petroleum-based diesel;

[0023] (b) from about 5% to about 15% by volume of the biodiesel; and

[0024] (c) at least about 5% by volume of the ethyl levulinate.

[0025] In one embodiment, the disclosed diesel composition, designated as "BEL20" fuel, comprises:

[0026] (a) about 80% by volume of ultra low sulfur diesel (ULSD);

[0027] (b) about 13.3% by volume of the biodiesel; and

[0028] (c) about 6.7% by volume of ethyl levulinate (EL).

[0029] The BEL20 fuel exhibits all required performances within the range specified under the ASTM 0975 specification, as shown in TABLE 1. Thus, the BEL20 fuel is in compliance with the ASTM D975 requirement for the commercial diesel fuels in the United States.

[0030] It will be understood that the BEL20 fuel is one exemplary embodiment for the fuel compositions of the present disclosure, and one skilled in the arts could readily modify the compositions to achieve certain selected performances.

TABLE 1

PERFORMANCES ACCORDING TO THE ASTM D975 SPECIFICATION			
Properties	Test Method	ASTM D975 Specification	BEL20 Fuel Properties
Flash Point (° C.)	ASTM D-93	Min. 52	74
Distillation (° C.), 90% volume	ASTM D-86	282-338	303
Viscosity (CST), at 40°C	ASTM D-445	1.9-4.1	2.2
Ash Content (wt %)	ASTM D-482	Max. 0.01	0.001
Sulfur Content (ppm)	ASTM D-5453	Max. 500	15
Copper Corrosion	ASTM D-849	3A	1A
Cetane Number	ASTM D-613	Min. 40	46
Cetane Index	ASTM D-976/4737	Min. 40	43
Aromaticity (vol %)	ASTM D-5186	Max. 35	31
Cloud Point (° C.)	ASTM D-2500	Report	-12
Low temp filter test/ Cold filter plug point (° C.)	ASTM D-6371	Report	-18

TABLE 1-continued

PERFORMANCES ACCORDING TO THE ASTM D975 SPECIFICATION			
Properties	Test Method	ASTM D975 Specification	BEL20 Fuel Properties
Carbon Residue mcr (vol %)	ASTM D-4530	Max. 0.15	0.098
Lubricity HFRR (microns)			
Major Axis	ASTM D-6079	Max. 520	460
Minor Axis			420
Wear Scar			440

[0031] The following examples are provided to further illustrate the present disclosure and are not to be construed as limiting the disclosure in any manner.

Experiments

[0032] Ethyl levulinate (EL) was used as a levulinate ester for the study. The Deutz 20114-cylinder, 3.11-1 turbocharged diesel engine was used for the testing. Three diesel fuels were used as fuel controls: ULSD diesel fuel, biodiesel fuel (BD), and "B20" fuel which is recognized as an alternative fuel under EPACT. A series of fuel blends were prepared as shown in TABLE 2. The properties of these blends, such as fuel consumption, performance and emissions, were investigated and compared to those of the three fuel controls.

TABLE 2

Tested Fuel	% Component (by % volume)		
	% ULSD	% Biodiesel	% Ethyl Levulinate
ULSD (Control)	100	0	0
B100 (Control)	0	100	0
B20 (Control)	80	20	0
EL5	95	0	5
BEL100	0	66.67	33.33
BEL20	80	13.33	6.47

Fuel Consumption

[0033] The fuel consumption (FC) of each fuel was determined at an engine speed of 2800 rpm and four different torque levels (150, 113, 75 and 29 lb-ft). The fuel consumption values of each tested fuel were compared to those of the control ULSD fuel at the same engine operation conditions. Percentages of the change in fuel consumption (% Change in Fuel Consumption) were calculated using the following equation:

$$\% \text{ Change in Fuel Consumption} = \frac{\left[\frac{FC \text{ of Tested Fuel} - FC \text{ of ULSD}}{FC \text{ of ULSD}} \right] \times 100}{FC \text{ of ULSD}}$$

[0034] FIG. 1 was a graph showing the percentage changes in fuel consumption of each fuel blend at different engine torques. On-road fuel consumption depends on the speed and torque of the engine. The study showed that when the engine was run at the high speed of 2800 rpm and low torque, the fuel consumption of BEL20 fuel blend was as good if not better than that of ULSD control.

Emission of the Oxide of Nitrogen (NOx) Gas

[0035] A Pierburg CLD PM 2000 chemiluminescence analyzer was used to measure the emission of NOx. The engine was run at a speed of 2800 rpm, and the NOx emissions for BEL20 fuel blend were determined at four different torque levels (150, 113, 75, and 29 lb-ft). The NOx emission of BEL20 fuel was compared to those of the control ULSD and B20 fuels at the same engine operation conditions.

TABLE 3

Tested Fuel	NOx Emission (g/hr) at Different Engine Torque (T)			
	T = 150 lb-ft	T = 111.3 lb-ft	T = 75 lb-ft	T = 29 lb-ft
ULSD	60.71	37.9	22.0	7.5
B20	61.8	38.6	22.0	7.3
BEL20	60.6	37.9	21.6	7.2

[0036] As showed in TABLE 3, EL fuel did not enhance the NOx emission of ULSD fuel as biodiesel did when the engine was operated at high torque values. The study showed that BEL20 fuel did not emit higher level of NOx compared to ULSD regardless the engine torque level.

Emission of Particulate Matters (PM)

[0037] All diesel particulate matter (PM) samplings were conducted using a Sierra Instruments' Model BG-2 Micro-Dilution Test Stand. The PM emissions for each fuel blend were determined using the ISO 8178 8-Mode Test protocol, and compared to the PM emissions of ULSD and B20 fuel controls. The PM emissions were measured for both volatile PM and non-volatile PM. The changes of PM (volatiles and non-volatiles) emissions for each fuel compared to the PM emissions for ULSD were calculated and showed in TABLE 4.

TABLE 4

Tested Fuel	Volatile PM		Non-Volatile PM		Total PM	
	Amount (g/bhp-hr)	% Change to ULSD	Amount (g/bhp-hr)	% Change to ULSD	Amount (g/bhp-hr)	% Change to ULSD
ULSD	0.065	—	0.079	—	0.144	—
B20	0.058	11%	0.061	-23%	0.119	-17%

TABLE 4-continued

Tested Fuel	Volatile PM		Non-Volatile PM		Total PM	
	Amount (g/bhp-hr)	% Change to ULSD	Amount (g/bhp-hr)	% Change to ULSD	Amount (g/bhp-hr)	% Change to ULSD
EL5	0.086	32%	0.059	-25%	0.146	1%
BEL20	0.059	-9%	0.044	-44%	0.103	-28%

[0038] As shown for B20 fuel in TABLE 4, addition of biodiesel to ULSD fuel led to a decrease in emissions of both volatile PM and non-volatile PM. As shown for EL5, addition of EL to ULSD fuel did not provide any significant change in PM emissions because the reduction in nonvolatile PM emission was offset by the increase in volatile PM emission. BEL20 fuel was a blend of EL with ULSD and biodiesel fuels. BEL20 fuel showed a substantially lower PM emissions compared to ULSD fuel (a 28% reduction in total PM emission). In particular, the non-volatile PM emission of BEL20 fuel was almost 45% lower than that of ULSD fuel. This unexpectedly high level of non-volatile PM emission reduction suggested a synergistic effect between EL and biodiesel in lowering the non-volatile PM emission of the ULSD fuel blend.

Cetane Number Study

[0039] EL has a lower cetane number than ULSD fuel and biodiesel. Therefore, addition of EL to the fuel blend of ULSD and biodiesel fuels reduced the cetane number of the resulting fuel blend. EL's higher autoignition point determines its low cetane number (CN=15) with longer ignition delays. It would be expected that the cetane number of the blend would be a weight average of the cetane number of the components. According to FIG. 2, the effective cetane number of the EL appeared to increase at higher blend levels, unlike that with the diesel which had no relationship.

Stationary Burner Study

[0040] EL was tested as a fuel in stationary burners, which was applicable to home heating and industrial burners. The commercially available Diesel No. 2 fuel was used as a control diesel fuel. The amount of sulfur dioxide (SO₂) and NOx gases emitted from the combustion of EL were measured and compared to those emitted from the combustion of Diesel No. 2 fuel. Furthermore, the combustion efficiency of EL was determined and compared to that of the Diesel No. 2 fuel.

[0041] TABLE 5 showed that compared to the diesel No. 2 fuel, EL provided a substantially lower SO₂ emission (97% lower) and NOx emission (67% lower), while providing about the same combustion efficiency.

TABLE 5

	SO ₂ Emission (ppm)	NOx Emission (ppm)	% Combustion Efficiency
Diesel No. 2 Fuel	61	119	86.56%
EL	2	39	86.2%
Difference	-97%	-67%	-0.5%

Lubricity

[0042] A series of diesel fuel blends were tested for lubricity HFRR according to the ASTM D-6079 standard method, as shown in TABLE 6.

TABLE 6

Tested Fuel	Lubricity HFRR (microns)
ULSD (Winter Formula)	535
100% Biodiesel	314
100% EL	285
B20 (80% ULSD, 20% vol biodiesel)	460
EL5 (95% ULSD, 5% vol EL)	405

[0043] The fuel with lower lubricity HFRR value provides superior lubricity property. According to the ASTM D-975, the diesel fuel must have the viscosity HFRR of no more than 520 microns. As shown in TABLE 6, EL5 fuel showed a significantly lower lubricity HFRR value than B20 fuel (405 microns vs. 460 microns) even when the % volume of EL was only 5% in the EL5 fuel blend compared to the % volume of biodiesel in B20 of 20%. Therefore, EL was far superior to biodiesel in enhancing the lubricity properties of ULSD fuel.

Cloud Point Temperature

[0044] A fuel blend was prepared containing 70% by vol ULSD fuel (winter formula), 20% by vol biodiesel, and 10% by vol EL. The Cloud point of the resulting fuel blend was determined and compared to those of the two control fuels: ULSD (winter formula) and B20 fuels. As shown in TABLE 7, the resulting fuel blend containing EL showed approximately the same cloud point as the ULSD fuel.

TABLE 7

Tested Fuel	Cloud Point (° C.)
ULSD (Winter Formula)	-10
B20 (80% ULSD, 20% vol biodiesel)	-7
Fuel Blend (70% ULSD, 20% biodiesel, 10% EL)	-10

Miscibility

[0045] TABLE 8 showed that EL exhibits good solubility in ULSD fuel, biodiesel, and B20 fuels.

TABLE 8

Tested Fuel	Solubility of EL in the Tested Fuel
ULSD (Winter Formula)	24%
Biodiesel	100%
B20 (80% ULSD, 20% vol biodiesel)	54%

[0046] While the disclosure has been described by reference to various specific embodiments, it should be understood that numerous changes may be made within the spirit and scope of the inventive concepts described. It is intended that the disclosure not be limited to the described embodiments, but will have full scope defined by the language of the following claims.

1. A fuel composition, comprising:
 - (a) from about 60% to about 80% by volume of the petroleum-based diesel;
 - (b) from about 5% to about 15% by volume of the biodiesel; and
 - (c) at least about 5% by volume of the levulinate ester.
2. The fuel composition of claim 1, characterized by performances in compliance with ASTM D975 Specification.
3. The fuel composition of claim 1, comprising less than 1% by volume of chemical including a member selected from a member selected from the group consisting of alkanolamides, alcohols, or combinations thereof.
4. The fuel composition of claim 1, wherein the petroleum-based diesel includes a member selected from a member selected from the group consisting of diesel fuel oil No. 2, diesel fuel No. 4, petroleum distillates, syntroleum, water-alcohol emulsified diesel, ultra low sulfur fuels, and blends thereof.
5. The fuel composition of claim 1, wherein the levulinate ester includes a member selected from a member selected from the group consisting of methyl levulinate, ethyl levulinate, butyl levulinate, glycol ester of levulinic acid, and blends thereof.
6. The fuel composition of claim 1, comprising:
 - (a) from about 60% to about 80% by volume of the petroleum-based diesel;
 - (b) from about 5% to about 15% by volume of the biodiesel; and
 - (c) at least about 5% by volume of ethyl levulinate.
7. The fuel composition of claim 6, characterized by performances in compliance with ASTM D975 Specification.

8. The fuel composition of claim 1, comprising:
 - (a) about 80% by volume of ultra low sulfur diesel;
 - (b) about 13.3% by volume of the biodiesel; and
 - (c) about 6.7% by volume of ethyl levulinate.

9. The fuel composition of claim 8, characterized by performances in compliance with ASTM D975 Specification.

10. The fuel composition of claim 1, further comprising an additive selected from a member selected from the group consisting of detergents, octane boosters, cetane number improvers, metal deactivators, corrosion inhibitors, antioxidants, rust inhibitors, dispersants, biocides, and combinations thereof.

11. A fuel composition, comprising:

- (a) a petroleum-based diesel; and
 - (b) at least about 5% by volume of levulinate ester based on total volume of the fuel composition,
- wherein the fuel composition includes less than 1% by volume of chemical including a member selected from a member selected from the group consisting of alkanolamides, alcohols, or combinations thereof.

12. The fuel composition of claim 11, further comprising biodiesel.

13. The fuel composition of claim 11, characterized by performances in compliance with an ASTM D975 Specification.

14. The fuel composition of claim 11, wherein the petroleum-based diesel includes a member selected from a member selected from the group consisting of diesel fuel oil No. 2, diesel fuel No. 4, petroleum distillates, syntroleum, water-alcohol emulsified diesel, ultra low sulfur fuels, and blends thereof.

15. The fuel composition of claim 11, wherein the levulinate ester includes a member selected from a member selected from the group consisting of methyl levulinate, ethyl levulinate, butyl levulinate, glycol ester of levulinic acid, and blends thereof.

* * * * *